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The Origin, Nature and Role of Naturally Occurring Organic Solutes In Unconfined Groundwater Systems.

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Introduction

In recent years there has been an increasing public awareness and concern about the quality of our groundwater resources. Of particular concern is the dramatic rise in the number of toxic and resistant man-made organic chemicals identified in groundwater. These organic contaminants may enter a groundwater system from a variety of sources; landfills, disposal compounds, chemical spills, sewage, burial sites, or by the application of pesticides and herbicides in agricultural practices.

Whereas considerable effort has been spent in the detection and remediation of man-made organic contaminants in the hydrosphere, and towards the establishment of drinking water guidelines, very little is actually known of the origin, geochemistry, and role of naturally occurring dissolved organic compounds (DOC) in groundwater. In fact, relatively few studies have addressed the inherent characteristics of "natural" DOC in groundwater (Leenheer et al., 1974; Barcelona, 1984; Thurman, 1985). Several studies, however, have demonstrated the importance of natural DOC as facilitating the transport of metals (McKnight et al, 1983), enhancing the solubility of insoluble organic contaminants (Perdue, 1983), and providing a carbon source for microbial redox processes (Miller et al.,1979; Starr et al., 1987).

Objectives

The overall purpose of this M.O.E. funded two year project (1987-1989) is to determine the origin and innate characteristics of naturally occurring organic solutes in groundwater, and to define the role of naturally occurring organic solutes in the interrelated biogeochemical redox processes that affect the carbon, sulfur, and nitrogen cycles of several shallow groundwater systems in Ontario.

Origin and Nature DOC in Groundwater

Dissolved organic carbon is widespread in groundwater, although typically at concentrations below 1-2 mg C/L (Leenheer at al., 1974). High molecular weight aquatic humic substances generally account for about 20-40% of the DOC in most groundwaters (Thurman, 1985), however, the nature of the remaining 60-80% of DOC remains largely unknown. In most aquifers there are two potential sources of DOC; organic leachate translocated via aquifer recharge from the soil and vadose zone, and/or organic matter derived from kerogen originally incorporated into the aquifer sediments.

This research will attempt to characterize and describe the geochemical evolution of high and low molecular weight organic compounds that form part of the DOC along the flow paths of several shallow aquifers. Evaluation of the age, origin and residence time of DOC in groundwater will also be assessed. Methods used are liquid chromatography, molecular sieving, stable isotopic mass spectrometry, gas chromatography/mass spectrometry (GC-MS), and Tandem Accelerator Mass Spectrometry (TAMS).

Role of Natural Organic Solutes in Groundwater

In anoxic groundwater systems DOC is thought to be the electron donor for the natural remediation of several prevalent inorganic contaminants, such as fertilizer derived nitrate and acid rain derived sulfate. Denitrification and sulfate reduction can affect the carbon cycle through the oxidation of organic matter and the concomitant production of respiratory carbon dioxide. However, in order for microbes to be able to utilize DOC to reduce NO_3^- or $SO_4^=$, the carbon source must be labile. Thus, the lability of DOC must be a function of the nature of the reduced carbon forms in the groundwater system. Extensive characterization of natural DOC of several groundwater systems in this study is expected to provide insight as to the "lability" of the various fractions of DOC. High DOC concentrations in groundwater may also be linked to microbial production of methane gas (eg. Aravena et al., this volume).

In addition, oxidation of DOC during microbial redox processes could have a potentially significant effect on the bicarbonate ^{14}C dating of groundwaters, especially if the organic carbon contains little radiocarbon. Thus, a better understanding of the role of DOC in groundwater is necessary to the interpretation of groundwater ^{14}C dating.

Other aspects of this research will attempt to address the effects of dissolved organic acids and anions on routine geochemical measurements, such as pH electrode determinations and alkalinity titrations of groundwater.

Site Selection

Three unconfined sand aquifers were chosen as field study sites. At all sites the groundwater flow regimes are reasonably well known, and previous groundwater surveys at these sites provide an additional degree of information on

geochemical processes in the aquifers (eg. Starr et al., 1987; Robertson and Cherry, submitted 1987).

Rodney Sand Aquifer

This unconfined, four meter thick sand aquifer underlies agricultural land near Rodney, Ontario, and comprises the glaciolacustrine sands of glacial Lake Warren. The site was used in a denitrification study by Starr et al. (1987). The water table is shallow (<100 cm), and significant concentrations of nitrate are rapidly denitrified within 50 cm below the water table. DOC in this system was presumed to be "labile", yet insufficient as the only carbon source for denitrification. The origin, fluxes, character and role of apparently "labile" DOC in this system is the focus of research.

Alliston Sand Aquifer

The unconfined, sand aquifer near Alliston, Ontario, was also used in previous denitrification studies (Starr et al., 1987). The aquifer underlies agricultural land, and is composed of glaciofluvial sands that form the present Nottawasaga River flood plain. The aquifer is hydrogeologically similar to Rodney, except that the water table occurs at a greater depth of 4 m. High levels of nitrate occur throughout this aquifer. Although DOC concentrations are similar to the Rodney site, no denitrification occurs even at 20 m depth. The origin, nature and role of seemingly "non-labile" DOC in this system is the focus of research at this site.

Sturgeon Falls Sand Aquifer

This unconfined, silty-sand aquifer is located on a forested post-glacial delta near Sturgeon Falls, Ontario.

Penetration of "bomb" produced radioactive Tritium into this system allowed for unusually accurate dating of groundwater recharge with a resolution of about 2 years spanning over 60 years of recharge (Robertson and Cherry, submitted 1987). This acid rain derived $SO_4^=$ which is prevalent in this system, however, undergoes dissimilatory sulfate reduction only after 16 m along the flow path (Robertson and Cherry, submitted 1987b).

The excellent hydrochemical resolution at this site is expected to yield detailed information about the temporal and spatial geochemical evolution of DOC, allow for accurate carbon mass transfer modelling, and also provide insight into the nature of carbon-sulfur cycle interactions.

Analytical Approach

All three sites have been previously instrumented with either stainless steel or PVC piezometers, spaced between 25 cm and 2 m apart. The piezometer nests are presently being sampled up to two times per year for complete water chemistry and environmental isotopes ($\delta^{18}O$, $\delta^{13}C$, $\delta^{2}H$, $\delta^{34}S$) Comprehensive geochemical measurements aid in the interpretations of basic hydrology and geochemical modelling. Statistical analysis of geochemical and DOC data will be used to test possible links between inorganic geochemistry and the organic carbon pool. Essential to DOC research in groundwater systems are a number of techniques adapted from marine and soil methods described below.

DOC Determinations

Reliable DOC determinations are essential for this study. Groundwater samples are carefully filtered through 0.45 micron filters and stored in glass vials to eliminate possible contamination. All samples are analyzed on a

calibrated Dohrmann Carbon Analyzer using standard techniques.

Liquid Chromatography

XAD-8 macroporous resins are used for the isolation of the aquatic humic substances from the groundwater, for determining the hydrophobic/hydrophilic fractions of the DOC; and for classification of the DOC (cf. Thurman and Malcolm, 1981; Leenheer and Huffman, 1979). Aquatic humic substances isolated by this method are used for carbon isotope determinations (^{13}C, ^{14}C).

Molecular Sieving

Silicalite™ molecular sieve is used to concentrate the low molecular weight ($<C_{20}$) portion of the hydrophilic fraction of DOC from the groundwater. DOC isolated in this molecular sieve is then further processed for GC-MS identification and for carbon isotope determinations (^{13}C, ^{14}C).

Gas Chromatography/Mass Spectrometry

A Hewlett-Packard GC-MS is used to assist in the identification of low molecular weight organic compounds adsorbed into Silicalite. Only the 20-30 most abundant GC peaks are evaluated by computer to identify specific organic compounds.

Stable Isotope Mass Spectrometry

A Micromass(TM) 903 VG mass spectrometer for determinations of stable isotope ratios is used to measure the ^{13}C content of DOC. This technique is an excellent tool for distinguishing between marine or terrestrial derived

DOC. Stable carbon isotopic ratios are also used as a tracer of both diagenetic processes and microbial activity affecting DOC.

Radiocarbon Dating

The recent development of a Tandem Accelerator Mass Spectrometer at the University of Toronto enables radiocarbon age dating of microgram size organic samples by direct counting of ^{14}C atoms. This new technology allows age dating of very small DOC samples (5-10 mg) isolated from groundwater using XAD-8 chromatography and Silicalite. Radiocarbon ages of DOC fractions are an excellent tool for determining the origin, age, and flux of DOC in groundwater systems. Radiocarbon dating of fractions of DOC in groundwater may also be a potential tool for the age dating of groundwater (Wassenaar et al., submitted).

Potentiometric Titrations

Potentiometric titrations of humic and fulvic acids extracted from groundwater are used to quantify carboxyl functional groups and phenol functional groups.

UV Absorbance

E_4/E_6 ultraviolet absorbance ratios of aquatic humic and fulvic acids extracted from groundwater are believed to provide an indication of molecular weight of aquatic humic substances. The E_4/E_6 ratios can be compared with soil and marine humic substances.

Elemental Analyses

The carbon, oxygen, nitrogen, hydrogen and sulfur content of aquatic humic substances extracted from

groundwater can be compared with those of humic substances from soils and marine environments. Thurman (1985) suggests that humic substances from groundwater contain less oxygen due to microbial action on DOC.

Preliminary Results and Discussion

Preliminary results of DOC determinations at all three sites are summarized in Figure 1 and 2. DOC concentrations in the groundwater at Rodney range between 1 and 12 mg C/L with a median of 4.6 mg C/L (Fig. 2). Generally, higher DOC contents (4-12 mg C/L) occur near the water table and decline with depth (1-1.7 mg C/L). DOC concentrations in the groundwater at Alliston range between 0.9 and 7 mg C/L, whereas, DOC in the groundwater at Sturgeon Falls ranges between 1.4 and 3.2 mg C/L (Fig. 1). These DOC concentrations are significantly higher than those observed in many deep confined aquifers (<1 mg C/L; Leenheer et al, 1974). The higher DOC concentrations in these shallow unconfined aquifers are no doubt a result of a lesser degree of biodegradation and attenuation.

DOC concentrations at the Rodney site also exhibit temporal variations (Fig 2). Whereas higher DOC concentrations occur near the water table, DOC peaks also occur below the water table. DOC peaks occur at 125 cm (12 mg C/L) and 225 cm (5 mg C/L) in April, 1988, and at 175 cm (4.5 mg C/L) for March, 1987. These peaks could represent the influx of spring or fall recharge, carrying high DOC waters leached from the soil zone. Antweiler and Drever (1983) and Cronan and Aiken (1985) have observed that higher concentrations of DOC occur in soils during spring runoff when the soil is saturated.

Starr et al (1987) measured DOC concentrations in the vadose zone from squeezed core material at this site. Their results are also summarized in Figure 2. The highest vadose/soil zone DOC values occur in the upper soil zone

with a maximum value of 150 mg C/L. From 30 cm to 90 cm depth, DOC drops to between 25 and 19 mg C/L and then to less than 12 mg C/L below the water table (Fig 2). The sharp drop in porewater DOC concentrations below 5-10 cm is a result of microbial biodegradation and adsorption in the vadose zone (Dawson et al, 1981; Meyer and Tate, 1983).

Nature of DOC

Preliminary results of the two main fractions of DOC isolated from groundwater at Rodney and Alliston are summarized in Figure 3. At Rodney the amount of dissolved hydrophobics varied between 15 and 88 percent of the total DOC, and showed significant variations with depth (Fig 2). Similarly, the hydrophilic fraction also varied with depth to between 12 and 85 percent of the total DOC. At Alliston the amount of hydrophobic DOC ranges between 0 and 50 percent of the total DOC. The hydrophobic fraction of DOC in these aquifers was determined to be >95% fulvic acid.

Preliminary data from the Rodney field site suggests the amount of DOC and its fractions may be temporally and spatially variable in aquifer recharge environments due to seasonal variations associated with groundwater recharge.

Hydrologic transport, however, may account for only part of the variation in the hydrophilic/hydrophobic fraction of DOC in groundwater. In the aquifers at Rodney and Alliston the hydrophobic fraction of the DOC shows a strong correlation with the total concentration of DOC (r^2 = 0.96; Fig 3). However, the hydrophilic fraction does not correlate as strongly with total concentration of DOC (r^2 = 0.52). Thus, changes in DOC concentration observed with depth are primarily due to changes in the hydrophobic fraction, and to a lesser extent the hydrophilic fraction.

GC-MS and carbon isotopic characterization of the DOC at the Rodney site suggest that microbial processes may also be an important control over the hydrophilic fraction of the

DOC. Table 1 lists compounds that we were able to identify by GC-MS for the hydrophilic fraction. The GC-MS chromatogram indicated the hydrophilic fraction at 125 cm depth was composed mainly (estimated >95%) of very low molecular weight compounds (C_4-C_1). The largest GC peak was identified as methyl acetate. The GC-MS used was not able to positively identify smaller organic compounds, such as formate. Acetate, however, is a well known microbial by-product of anaerobic fermentation of kerogen and perhaps DOC, and is the most common volatile fatty acid found in natural waters (Miller et al, 1979; Barcelona, 1980; Thurman 1985). Interestingly, methyl acetate, as well as many other LMW compounds identified at 125 cm (DOC=12 mg C/L), are not present at a depth of 250 cm (DOC = 4 mg C/L; Table 1). We believe many of these LMW compounds may have been utilized by microbes in the zone of denitrification and sulfate reduction that occurs between 100 and 200 cm depth. LMW organic acids, and especially acetate are well known substrates for nitrate, sulfate reducing and methanogenic bacteria (cf. Cappenberg and Prins, 1974). It is possible, however, that seasonal variations in DOC recharge may also affect the types of LMW compounds entering the aquifer.

Virtually all LMW compounds identified in the hydrophilic fraction of the DOC at Rodney (Table 1) are potential degradation products of lignins, cellulose, and soil organic matter (Thurman, 1985). The two chlorinated compounds identified, however, may possibly be the result of a reaction of chlorine (from chlorinated herbicides?) and fulvic acid (cf. Oliver and Visser, 1980), and/or derived from degradation of herbicides and fertilizers applied to the field.

The data also suggest that some U.S.A. EPA rated priority pollutants (eg., toluene, benzene, xylenes) occur naturally at trace levels in groundwater, although it was not possible with the isolation method used to quantify their concentrations. Our research suggests that further

data on "natural" levels of toxic organic compounds in groundwater should be obtained. A separation between "natural" and anthropogenic organic compounds may provide better criterion for the establishment of drinking water guidelines.

Carbon Isotopes and DOC

Preliminary results of $^{13}C/^{12}C$ and ^{14}C TAMS analyses of the hydrophobic and hydrophilic fractions of the DOC from the Rodney aquifer are summarized in Figure 2. Carbon-13 isotope values measured for both hydrophobic DOC samples were -27.3 °/oo and -30.5 °/oo (PDB; Fig 2). These values are similar to soil organic matter and humic substances derived from C_3-type vegetation (Deines, 1980). The $\delta^{13}C$ value of the hydrophilic fraction at 125 cm, however, has an extremely depleted value of -47 °/oo (PDB). If one assumes that the hydrophilic fraction at 125 cm is a result of microbial fermentation of particulate organic matter and/or DOC, as is suggested by the presence of methyl acetate in the sample, then microbial isotope effects may be the cause for ^{12}C enrichment in the sample. This carbon isotopic fractionation process may be analogous to the large isotope effects observed in microbial methane fermentation (cf. Deines, 1980; Barker and Fritz, 1981).

A loss of ^{12}C enriched LMW acids is evident below the nitrate and sulfate reduction zone at Rodney. At 250 cm depth only larger molecular weight (>C_6) organic compounds were identified and the sample had a $\delta^{13}C$ value of -24.0 °/oo, which is similar to that of the precursor C_3 plant matter (Deines, 1980). It is possible that LMW organic compounds, such as acetate or formate, are preferentially utilized by nitrate and sulfate reducing microbes in groundwater.

Radiocarbon data for the hydrophobic and hydrophilic fractions of the DOC at Rodney are also summarized in Figure

2. The results indicate significant differences in ^{14}C activity between hydrophobic samples collected at 125 and 250 cm depth. (NOTE: ^{14}C activity is usually expressed as percent modern carbon- PMC, or as a conventional age in years before 1950 - B.P.) Substantial differences in ^{14}C activity are also observed between the hydrophobic and hydrophilic fractions. The hydrophobic fraction at 125 cm has a ^{14}C activity of 91.8 PMC (600 years B.P.), but has only 76 PMC (2,200 years B.P.) at 250 cm (Fig 2). Clearly, the hydrophobic fraction at 125 cm (91.8 PMC) must originate from the upper soil profile when compared to the ^{14}C activity of humic substances extracted from a core, which are only 79 PMC (1,910 years B.P.) at a depth of 30 cm (Fig 2).

Considering the hydrology of the Rodney aquifer, the reduction in ^{14}C activity of the hydrophobic fraction cannot represent the advective residence time between 125 and 250 cm. Two theories may account for this lower ^{14}C activity of the hydrophobic fraction at 250 cm. One possibility is that the hydrophobic fraction of the DOC is a mixture of young carbon from the soil zone and older components from the sediments, the latter being more significant at depth. This mixing of young and older humic substances may be seasonally variable. However, one can also argue that perhaps a "younger" (and more labile?) component of the hydrophobic fraction of the DOC is being preferentially consumed by microbes. It is generally believed that humic substances and aquifer kerogen, due to their refractory nature, are not a readily available carbon source for microbes. If this is true then the addition of large molecular weight humic substances from aquifer organic material would not account for the observations.

A second possibility is that the ^{14}C activity differences of the hydrophobic fraction in the aquifer as a whole simply reflect temporal differences in the ^{14}C activity of DOC entering the aquifer from the vadose zone

(ie, spring DOC flux vs. fall recharge). In this respect it is interesting to note that the lower ^{14}C-DOC activity at 250 cm depth is paralleled by a $\delta^{18}O$ value of the water near -11 °/oo reflecting winter/spring recharge, whereas the sample collected at 125 cm has a $\delta^{18}O$ value near -8 °/oo reflecting summer/fall recharge.

The ^{14}C activities of the hydrophilic fraction are significantly lower, 67-69 PMC (2930-3280 years B.P.), than the hydrophobic fraction and are similar for both samples (Fig 2). This requires a significant input of old carbon to the hydrophilic fraction of the DOC.

We have identified a large component of LMW organic acids for the sample at 125 cm at Rodney (Table 1). The presence of methyl acetate, and possibly the ^{12}C enrichment, may indicate this fraction of the DOC contains a significant amount of organic solutes derived from microbial fermentation of kerogen and/or DOC. This fraction does not correlate strongly with total amount of DOC (Fig 3), which has been shown to vary seasonally, and is generally present in concentrations of less than 2 mg C/L. The similarity of the ^{14}C content of the hydrophilic fraction of the DOC and the carbon in the solid phase (Fig 2) seems to suggest a direct link between the two components.

Current Research

Completion of field research and data analysis will comprise the final year of this project (1988-1989). Our immediate goals are;

1. to complete isolation of natural DOC at Alliston and Sturgeon Falls for geochemical analyses,
2. to complete isotopic and GC-MS analyses of DOC at Alliston and Sturgeon Falls sites,
3. to complete detailed characterization of aquatic humic substances from all three sites,

3. to identify redox processes that utilize DOC and affect the C, S, and N cycles of these aquifers,
4. to present a cognitive model for the origin and age of natural organic solutes in different groundwater regimes, and,
5. to present a quantitative geochemical model for the evolution and mass transfer of DOC in groundwater flow systems.

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Table 1. GC-MS identified dissolved hydrophilic compounds from Rodney sand aquifer. Samples were thermally desorbed from silicalite molecular sieve.

R3-1 - 125 cm	R3-6 - 250 cm
Dominant Compounds	
methyl acetate	none
Minor Compounds	
toluene	n-hexane
hydroxylamine	p + m xylenes
chloroform	cyclohexane
2-ethyl-1-hexanol	cyclopentane
p + m xylenes	stearic acid
diphosphoric acid, diisooctyl ester	palmitic acid
1,2-dichloro-benzene	
2-methyl propanate	
2,6-dimethyl-nonane	
benzene	
ethyl benzene	
benzoic acid, methyl ester	

List of Figures

Figure 1. Concentrations of natural organic solutes in two unconfined sand aquifers of Ontario. Triangles denote the Alliston site, and squares denote Sturgeon Falls field site.

Figure 2. DOC concentrations, percentage of hydrophobic and hydrophilic fractions of the DOC, weight percent organic solids, 14C activity and 13C (PDB) isotope values of DOC and organic solids from the Rodney sand aquifer. Sampling times are denoted by; □ - January, 1987, ○ - March, 1987, △ - June, 1987, * - April, 1988. Vadose zone data (▲) and weight % organic solid determinations (●) from Starr et al (1987). Note upper scale for vadose zone DOC concentrations. See preliminary discussion of data for details.

Figure 3. Correlation of the hydrophobic and hydrophilic fractions of DOC with total DOC concentration. Based on combined data from the Rodney and Alliston sand aquifer.

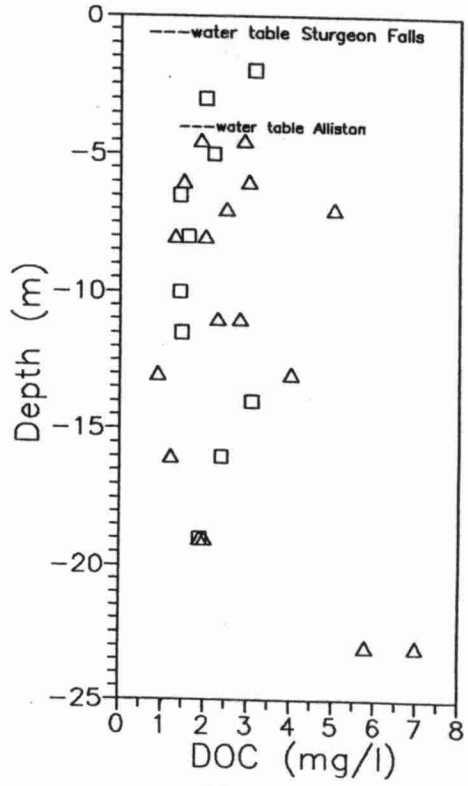

Figure 1

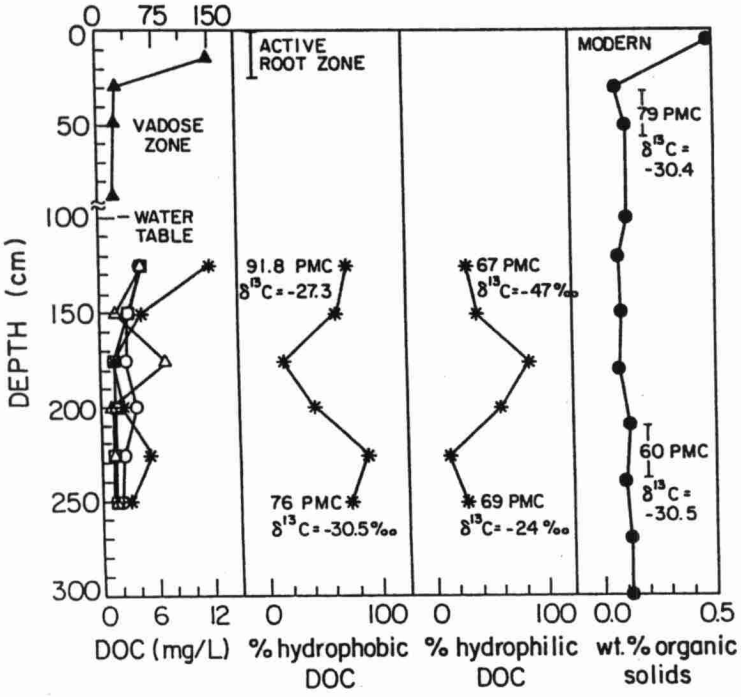

FIGURE 2

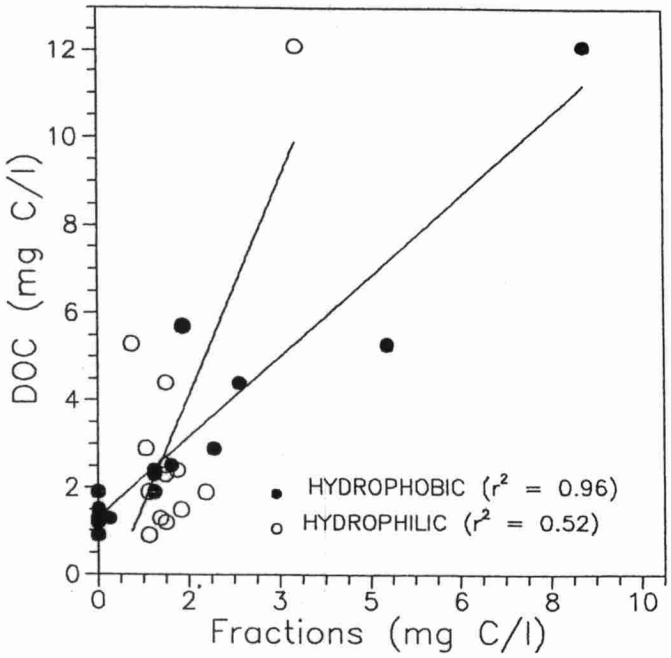

Figure 3

(8422)
TD/5/T43